Performance Monitoring Protocol (QA/QC) for the Raman Spectrometer

1 Scope

This document addresses the performance monitoring (QA/QC) of the Raman Spectrometers (sample compartment and/or microscope). This document applies to personnel using the associated instrument(s)/equipment in the following discipline/category of testing: Explosives (chemistry) examinations performed at the Huntsville facility.

2 Principle

A Raman spectrometer can be used to analyze samples in larger quantities in the sample compartment, if available, or in smaller quantities on the microscope, utilizing one or more objectives (e.g., 10X, 50X, 100X). In general, the signal for opaque samples can be maximized with a high numerical objective utilizing a microscope, while the signal for transparent samples can be maximized using a macro lens (i.e., sample compartment) or small magnification objective. The Raman spectrometer, either Fourier transform or dispersive, may utilize one or more excitation lasers (e.g., 1064 nm, 785 nm, 780 nm, 532 nm). In general, the signal will be more intense with a shorter wavelength excitation source; however, there is a trade off as samples may fluoresce and/or overheat with higher energy. Definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Protocol."

3 Equipment/Materials/Reagents

a. Thermo DXR Dispersive Raman Spectrometer System with sample compartment and microscope, Omnic Software (or equivalent)

b. Polystyrene slide, rod, or disk, such as FT-Raman standard (Thermo or equivalent)

c. Alignment tool containing calibration slide with pinhole and white light (Thermo or equivalent)

4 Standards and Controls

4.1 Performance Verification Standard

Polystyrene is used to assess daily operating performance and continued integrity of the Thermo Raman systems. Polystyrene requires no preparation and does not expire.

4.2 Alignment Tool

The alignment tool is used as needed to verify that the sample compartment and microscope are aligned and functioning properly. There is no sample preparation involved. The tool does not expire.

5 Sampling

Not applicable.

6 Procedures

6.1 Daily Checks

6.1.1 Microscope

The following steps are to be performed daily. Enter the appropriate information in the QA/QC log for tracking purposes.

a. Start Omnic and turn on the desired laser (if applicable) under 'Experiment Setup.' Allow time for the laser(s) to warm up and for the detector/ccd to cool. If the detector/ccd is not cool, a message will be displayed when data collection is attempted. The current temperature readout can also be viewed under the Advanced Tab in Experiment Setup. When the detector/ccd temperature setpoint has been reached, data collection can begin.

b. Turn the microscope illuminator on.

c. Set the operating parameters as listed in the 'Instrumental Conditions' section of this protocol.

d. Place the polystyrene slide on the stage and focus. Collect the sample spectrum. Perform a peak analysis by using the 'Find Peaks' option under the 'Analyze' menu. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the labeled spectrum.

e. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, contact the appropriate instrument support personnel.

6.1.2 Sample Compartment

The following steps are to be performed daily. Enter the appropriate information in the QA/QC log for tracking purposes.

a. Start Omnic and turn on the desired laser (if applicable) under 'Experiment Setup'. Allow time for the laser(s) to warm up and for the detector/ccd to cool. If the detector/ccd is not cool, a message will be displayed when data collection is attempted. The current temperature readout can also be viewed under the Advanced Tab in Experiment Setup. When the detector/ccd temperature setpoint has been reached, data collection can begin.

b. Set Operating Parameters as listed in the 'Instrumental Conditions' section of this protocol.

c. Place the polystyrene rod in the sample compartment. Collect the sample spectrum. Perform a peak analysis by using the 'Find Peaks' option under the 'Analyze' menu. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the labeled spectrum.

d. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, contact the appropriate instrument support personnel.

6.2 As Needed Checks

The following procedure shall be performed as needed based on performance. Indicate completion in the appropriate log.

a. Alignment of the Spectrometer:
Refer to the instrument manual.

b. System tuning (Microscope DXR Only):
Focus the alignment tool on the white light. Verify calibration by choosing 'Calibrate Instrument' under the 'Collect' pulldown and, if applicable, check the boxes for:
- Laser frequency calibration
- Wavelength calibration

c. If all requirements are within specification or a successful result is returned, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, contact the appropriate instrument support personnel.

7 Instrumental Conditions

Refer to the "General Instrument Maintenance Protocol" for procedures on minor deviations.

7.1 Microscope

Collect Tab
Set exposure time = 20 seconds
Set number of exposures = 2
Set final format - shifted spectrum (cm^{-1})
Cosmic ray rejection, to coincide with analysis set-up
Set correction to white light, if applicable
Set number of background exposures = 2

Bench Tab
Select microscope as 'Beam path/Accessory'
Select laser, to coincide with analysis set-up
Set laser power level to 100%
Select aperture, to coincide with analysis set-up
Select objective, to coincide with analysis set-up
Set resolution to same conditions as sample analysis
Set Grating Positions = multiple
Set maximum range limit to 3300 cm^{-1}, and minimum range limit to 200 cm^{-1}

7.2 Sample Compartment

Collect Tab
Set exposure time = 20 seconds
Set number of exposures = 2
Set final format - shifted spectrum (cm^{-1})
Check 'Cosmic ray rejection'
Set correction to white light
Set number of background exposures = 2

Bench Tab
Select '180-degree' as 'Beam path/Accessory'
Select laser, to coincide with analysis set-up
Set Focus, 'Side to side', and 'Up/down' parameters to achieve maximum signal
Set laser power level to 100%
Select aperture, to coincide with analysis set-up
Set resolution to same conditions as sample analysis
Set Grating Positions = multiple
Set maximum range limit to 3300 cm^{-1}, and minimum range limit to 200 cm^{-1}

8 Decision Criteria

- The Polystyrene spectrum is acceptable if all peaks are within ±5 cm^{-1} of the expected values, listed below (in cm^{-1}):

  621 796 1001 1032 1155 1451 1583 1602 2852 2905 3054

9 Calculations

Not applicable.

10 Measurement Uncertainty

Not applicable.

11 Limitations

Only properly trained personnel will perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

12 Safety

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal protective equipment should be used when handling any chemical and when performing any type of analysis.

13 References

Manufacturer's Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Protocol" (IOG 001) *Instrument Operations Group SOP Manual.*

DXR Manuals.

FBI Laboratory Safety Manual.

Rev. #	Issue Date	History
0	10/04/18	New document that specifies instrument protocol for the Huntsville facility.

Redacted - Signatures on File

Approval

Scientific Analysis
Unit Chief Date: 10/03/2018

TL Approval

Explosives (Chemistry)
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QA Approval

Quality Manager Date: 10/03/2018